

First Quarterly Report

STUDY OF NICKEL-CADMIUM CELLS

17 May 1965 - 17 August 1965

Contract No. NAS5-9586

Prepared by

General Electric Company
Research & Development Center
Schenectady, New York

GPO PRICE \$ _____

CFSTI PRICE(S) \$ _____

for

Hard copy (HC) 2.00

Microfiche (MF) 50

ff 653 July 65

NASA
Goddard Space Flight Center
- Greenbelt, Maryland

N66-14273

FACILITY FORM 602

(ACCESSION NUMBER)
26
(PAGES)
OR 68995
(NASA CR OR TMX OR AD NUMBER)

(THRU)
1
(CODE)
03
(CATEGORY)

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SUMMARY

The work to be performed under this contract consists of two phases; one, a detailed study of the behavior of the positive nickel hydroxide electrode, with respect to its charging characteristics, gassing behavior, and charge retention; and the other a study of the sources and effects of impurities on the performance of nickel-cadmium cells. This program is an extension of the work on the characterization of nickel-cadmium cells conducted under a previous contract NAS5-3477. The overall goal of this work is the development of nickel-cadmium cells with more uniform capacity and operating characteristics such as charge and discharge voltage, charge acceptance, pressure behavior during charging and cycle life.

The work during the first quarter consisted of assembly and testing of an oxygen monitoring system for use in the study of the positive electrode behavior and laying out work plans for the program. Receipt of electrodes from the Battery Business Section for use in the test program is scheduled for late September. Experimental work is scheduled to start on October 1, 1965.

1.0 INTRODUCTION

The results of the work performed under a previous contract (NAS 5-3477) on the characterization of nickel hydroxide and cadmium electrodes, as well as work performed by other investigators, show that in large part the variable or erratic behavior of nickel-cadmium cell can be assigned to the behavior of the nickel hydroxide electrode. A second major contributing factor is the presence of impurities introduced into the cell either during its manufacture or formed as degradation products from cell components during the operation of the cell.

The objective of the program is to determine the relative importance of these two factors and the extent to which the variability in cell performance can be reduced. The experimental work will include detailed studies of individual positive electrodes and cyclic sealed cell performance tests.

During the first quarter an experimental facility to measure the onset and the rate of oxygen evolution from positive electrodes was assembled and partially tested. The facility has a capacity for testing six electrodes simultaneously. The equipment has provisions for recording the rate of oxygen evolution, electrode potentials versus a reference electrode during charge and discharge. The equipment will permit the study of the positive electrode behavior over a range of charge and discharge rates on a repetitive cycle basis.

In addition to above work, literature pertaining to the effects of impurities on the nickel-cadmium system was reviewed and approaches and areas of emphasis for the impurity studies were selected.

2.0 PROGRAM SCOPE AND PLANS

An outline of the scope of the program is given in Table 2-1.

2.1 Positive Electrode Behavior Studies

The results of the previous work show that a large part of the erratic behavior of nickel-cadmium cells can be assigned to the behavior of the nickel hydroxide electrodes used in the cells. The causes for this erratic behavior are not well understood nor does the behavior appear to be predictable on the basis of previous cycle data or general history. Part of the erratic behavior must result from the high degree of nonreversibility of the electrode. The electrode is thermodynamically unstable with respect to oxidation of water in the presence of alkali, and only the high overvoltage for oxygen evolution on nickel and nickel oxide-hydroxide surfaces makes possible the charging and use of a nickel (III) hydroxide electrode.

The voltage of the positive electrode during charging is a function of the state-of-the-charge of the electrode and the previous cycling history of the cell. The voltage should show a marked inflection at the end of charge but does not. This is attributable to the formation of mixed nickel (II) and (III) hydroxides in the electrode during charging. These hydroxides are present as a single solid phase and therefore show only a mixed potential for the electrode. The electrode voltage therefore rises as the charging proceeds with no sharp break in the voltage curve at the end of the charge associated with the change from nickel (II) to nickel (III). Associated with this mixed potential of the plus two and plus three valence states of nickel, is the mixed potential associated with the evolution of oxygen from the surface of the electrode. The net result is that the charge voltage of the nickel-cadmium cell is more a function of the previous history of the cell than it is of the thermodynamic properties of nickel (II) and nickel (III) hydroxides. It is possible to make this charging potential fall within a narrow band of values by appropriate treatment of the electrode; it is not clearly understood how this treatment works. The voltage on discharge also shows variations depending upon cycle history which may be due again to changes in the electrode, particularly the electrode surface, brought about during the previous operation of the cell.

Table 2-1

PROGRAM SCOPE

- TASK 1: Study of the positive electrode behavior
- A. Voltage behavior during charge and discharge
 - B. Capacity relationships
 - C. Gassing behavior during charge, stand, and discharge
 - D. Mechanical aspects of overcharge behavior
- TASK 2: Study of the sources and effects of impurities in nickel-cadmium cells.
- A. Manufacturing impurities
 - 1. Electrolyte
 - 2. Plate
 - 3. Construction techniques
 - B. Cell degradation products
 - 1. Corrosion of components
 - 2. Decomposition of organic materials used in the cell construction

The capacity of a positive electrode shows three distinct types of capacity during normal operation of the cell. These types are: a permanent capacity which constitutes the greater part of the capacity of the cell that is obtained on discharge at cell voltages above one volt; a temporary capacity which is obtained at a somewhat higher voltage, but which is lost upon stand within a short period of time; and finally, a so-called graphitic capacity which is associated with a lower output voltage in the vicinity of 0.9 volt which is obtained at the end of discharge of the cell. The permanent capacity is only relatively permanent, as it is lost at a slow rate depending upon cell history, cell construction, and particularly temperature.

The temporary capacity appears to be associated with the charging of the permanent capacity by being made up of the initial active species formed on charging; part of this species reverts to permanent capacity upon stand,

and part is lost by reaction of the active species with the electrolyte to form oxygen. In any case, the temporary capacity is lost within 48 hours after shutting off the charging current; the loss may be as much as 20 percent of the total capacity delivered above one volt. A large amount of work has been done to determine the nature of the electrode material making up the temporary capacity and how to stabilize or convert it to permanent capacity. Very little success has been reported in this regard. The temporary capacity is a critical element in space applications in which the cells are charged and immediately discharged on short cycles since it contributes a large part of the capacity obtained from the cells during operation.

The graphitic capacity was given this name because it was formerly thought to be associated with the graphite that was incorporated in early designs of positive electrodes. However, this particular capacity persists even in the absence of graphite in the structure and is now attributed to adsorbed oxygen gas that is held by the lattice of the nickel sinter. This oxygen may be in the form of absorbed oxygen on the nickel substrate, or it may be in the form of a peroxide type of compound with nickel or cobalt. In any case, the discharge of this capacity occurs at a voltage substantially lower than for the normal capacity of the cell. Therefore it is not considered to be particularly useful as a capacity in the cell. The work on the previous contract showed that graphitic capacity in general decreased with cycle life. In a few cases the capacity increased.

In the proposed investigation of the capacity behavior of the nickel hydroxide electrode, the emphasis will be on obtaining data relating to the causes for the change of the graphitic capacity and its relation to change in permanent capacity. Emphasis will also be given to obtaining data relating to the stabilization and utilization of the temporary capacity in the discharge cycle for space applications. From the design standpoint, the change in capacity of the positive electrode with use is critical, since most space cells must maintain a careful balance between the negative and positive electrode capacities in order to promote gas recombination.

The gassing characteristics of the positive electrodes are highly irregular from cycle to cycle; they particularly vary with the previous history of the electrode. It is apparent that the process is highly irreversible and is subject to a high activation energy for the formation of molecular oxygen, and that a catalyst of some sort is involved in the electrode process evolving oxygen. This catalyst may be associated with the formation of nickel (III) oxide in the presence of the cobalt that is normally present in the electrode during fabrication. The onset of gassing occurs earlier in repeated cycles when the electrode is first cycled; finally coming into a region in which the initial gassing in a cycle occurs in a band of values between 40 to 60 percent of charge. The amount of gassing is also dependent to some extent on the previous history but varies from cycle to cycle in an irregular manner. The importance of the gassing behavior is that it represents a loss of charge acceptance and therefore must be known if a suitable charging regime for the cells is to be selected. In addition, the gassing characteristic determines to some extent the pressures that are going to be built up in the cell during charging operations which in turn determine some of the structural requirements of the cells. The actual work on gassing will be largely to determine factors involved in the gassing process and what, if any, impurities or other treatment can be used to make the gassing more uniform in cell operation.

The mechanical aspects of the positive electrode behavior are largely associated with overcharge of the electrode. During overcharge, oxygen is evolved from the electrode, and it has been found that this oxygen evolution exerts a profound influence on the structural stability of the nickel sinters in the electrode. The resistance to destruction by oxygen evolution varies from lot to lot of nickel sinters in a manner that is not directly related to any of the known process variables. The destruction appears to be caused by the formation of gas bubbles in the interior of the metal sinter which exert a large force as they grow by electrochemical formation of oxygen. These finally lift portions of the sinter structure from the substrate. It is not too uncommon to find electrode structures disintegrating in a short period of operation. However, using the same procedures for making these

electrodes and controlling the variables as closely as possible, other batches of electrode material show a relatively high resistance to this form of destruction. It therefore becomes important to determine what variable is responsible for the destruction of the electrodes in one case and not in the second, and also to determine the importance of this in terms of the life of a cell and its operation.

2.2 Impurity Study

There are two sources of impurities in sealed nickel-cadmium cells; one arising from manufacturing operations and the other arising from chemical attack on the material used in the cells during the operation of the cell. The impurities introduced by manufacturing techniques are in theory controllable by adequate quality control procedures; however, complete removal of some of these impurities would introduce prohibitive costs in manufacturing cells. Impurities such as dirt, welding flux residues, metallic chips, and dust, are beyond the scope of this study since these are impurities that can be completely eliminated with ordinary care.

However, there are a number of impurities that can be introduced into the cell during manufacturing that cannot be avoided except by taking exceptional care; in most cases, extra steps must be used in processing the materials in the cells to remove the impurity. Among these impurities are: nitrates and metallic impurities introduced into the plates during impregnation; and chlorides, sulfates, and carbonates introduced into the cell in the electrolyte. The effects of these impurities have been studied to some extent by previous workers; most of the work has been done on the effect of the impurities on the early behavior of the cell, with little emphasis on the effect on cell life.

At present, there is no good nondestructive method for the determination of nitrates in a cell. However, we have found that there is a plateau in the discharge curve of a positive electrode that corresponds to the reduction of nitrite to ammonia. This plateau is seen only on overdischarge of the

electrode at low rates; the area under the curve is proportional to the amount of nitrite reduced. This may serve as the basis for a nitrate analysis in the completed cell and will be investigated.

Work at the SAFT Research Laboratory, in France, has shown that sulfate and chloride are relatively innocuous as impurities in the levels that might be experienced in the electrolyte. They would occur in the electrolyte as a result of carrying over traces of the starting materials from which potassium hydroxide is made; namely, potassium chloride or potassium sulfate. Of more critical importance would be carbonate impurities which can be introduced as a contaminant from air. It should be noted that carbonate can be derived from oxidation of organic matter during operation of the cells so that the initial control of carbonate concentration in the electrolyte does not necessarily mean that no carbonate influence will be found in the cells at a later date.

The presence of carbonate affects the potential of the cadmium electrode when it replaces part of the hydroxyl ion in the electrolyte. As long as there is sufficient hydroxyl ion present, the cadmium electrode is unaffected at room temperatures and above, since cadmium hydroxide is much less soluble than the carbonate. However, if there is insufficient hydroxyl ion present, the cadmium precipitates as the carbonate, with a loss of about 0.1 volt on discharge. Carbonate has a particularly adverse effect at low temperatures. The drop in potential due to formation of cadmium carbonate is serious in cell applications where the cells are discharged to final cell potentials of one volt, as the apparent capacity will be decreased.

Small amounts of nitrate impurities in nickel-cadmium cells increase the self-discharge rate and also appear to decrease the capacity of the positive electrode. The reaction mechanism appears to vary with temperature and cycling conditions. In one case, the reaction appears to be the cyclic reduction of nitrate at the charged cadmium electrode followed by oxidation of nitrite at the charged positive electrode. In another case, there is evidence that the nitrate can be ultimately reduced to ammonia and eventually nitrogen. Which of the mechanisms prevails and under what conditions is

important since in the former case, the discharge reaction would prevail through out the use of the cell whereas in the latter, the effect is ultimately removed by cycling. This point will be studied in the current program.

Additional impurities can be introduced during manufacturing operations by the thermal decomposition of organic cell materials near the welds made to seal off the cell. Thus, thermal decomposition of the fluorothene cell liner used to insulate the electrode pack from the cell case would introduce fluoride and chloride. Thermal decomposition of any epoxy used to coat the underside of the cell terminals, or the thermal decomposition of any elastomer used to make the seals, would also introduce impurities in the cell system. In the case of epoxies, nitrogenous impurities could be introduced (nitrogen compounds are used as curing agents in most epoxy formulations) as well as carbonate and carboxylic that would be generated by oxidation of the epoxy carbon chain. Impurities from seal elastomers are probably not harmful. If the elastomer is heated sufficiently to give off injurious amounts of impurities the heating will probably result in a damaged seal that will not pass subsequent tests.

Metallic impurities such as iron, copper, zinc, etc., can be introduced into the plates during impregnation. Of these, iron is the most likely and is a serious impurity in positive electrodes since it seriously limits the deliverable capacity of the electrode. However, iron is very often added to the cadmium plate as an expander. Therefore, its presence in the cadmium plate is not of too much concern. The presence of iron as an impurity in the nickel hydroxide electrode can be due to the corrosion of the iron grids used in cell construction and to an impurity in the nickel nitrate used for impregnation. The presence of cobalt, which is common in commercial nickel salts, is not considered an impurity as it is deliberately added in many formulations for the positive electrodes. Previous work has shown that some impurities, such as magnesium and aluminum, have a promotional effect on the positive electrodes rather than a deleterious effect and therefore are of little concern.

The impurities introduced into the cell during operation arise from a chemical attack on the cell construction materials by oxygen and caustic.

Possible impurities that can be introduced in this manner include silver from the silver solder used to make hermetic seals, aluminum from the ceramic portions of the seals, iron from the case and electrode leads, nitrogenous materials from nylon separators, carbonate and carboxylic acids from separators, cell liners and wetting agents, and fluoride, chlorides and cyanides from separator or lining materials.

The effects of some of these impurities are known to a limited extent. Thus, silver has been reported to form dendrites in the cell during operation that result in shorting of the electrodes. Silver is known to be a catalyst for the oxidation of organic materials in the presence of alkali by oxygen. The oxidation of the separator material is known to occur in cells during their lifetime, even with resistant materials such as nylon and polypropylene. Oxidation of nylon would result in the formation of carbonates, carboxylic acids, and combined with hydrolysis induced by the alkali, ammonia or other nitrogenous derivatives. Oxidation of polypropylene would give carbonates and carboxylic acids; the oxidation of Dynel would give carbonates, carboxylic acids, and possibly cyanide or ammonia. The formation of some of the intermediate oxidation products from the separators is known to be beneficial probably because they enhance the wetting characteristics of the separator.

The origin and quantity of these impurities introduced into the cell will be determined in the program by exposing appropriate materials to systems simulating the cell environment: caustic electrolyte and free oxygen in the presence of nickel (III). Where possible, the nature of the impurity obtained by breakdown of organic materials will be determined by suitable analytical techniques and the amount of the impurity estimated. The effects of the impurities will be determined in cell tests.

2.3 Work Plans

The electrodes to be used in the program will be type VO positives and negatives made at the General Electric Company-Battery Business Section facility at Gainesville, Florida. Plate dimensions will be the same as used

in the previous program 1 3/4 x 3 inches. The positives will have a normal capacity of 1.0 ampere hours and the negatives 1.5 ampere hours. A lot of 1000 plates each of positives and negatives will be available for the program. These plates are scheduled for delivery in late September. Plate manufacturing history and characteristics will be available.

The plates will be given identification numbers and weighed as received. The lots will be sampled and the plates will be initially characterized by six charge-discharge cycles using a C/4 rate based on the nominal capacity of the positive plates, for both charge and discharge at ambient temperature. Complete potential-time curves versus reference electrode will be recorded for these cycles. A sufficient number of plates will be characterized in this manner to obtain a capacity distribution curve for the lots. Plates from the low, average and high regions of the capacity distribution curve will be used in the positive electrode behavior studies. This procedure will permit a determination of the effect of plate capacity on the positive electrode behavior. The characterization procedure will also permit the selection of plates with closely matched capacities for use in the fabrication of test cells for the impurities studies.

In the positive electrode behavior studies, the primary parameters to be investigated will be the effect of charging rates over the range of C/10 to C, the effects of repetitive charging at fixed rates versus variation in charge rate from cycle to cycle, and the effects of cyclic operation partially discharging of the positive electrode to various depths versus complete discharge. For the electrodes on partial discharge type cycling, the residual capacity will be periodically determined to note if memory effects are correlated with charge rate sequences, state of charge or gassing behavior. Self-discharge characteristics on open circuit will be monitored making gas evolution measurements along with voltage decay versus time from various states of initial charge.

In addition, the short term effects of impurities identified in Task 2 on the positive electrode behavior with respect to voltage, capacity and gassing rate will also be determined prior to cell tests. An attempt will be made to

relate observed voltage, capacity and gassing behavior variations to structural or chemical changes in the electrodes.

Areas of emphasis on the impurities studies will be to identify the sources and effects of nitrates, chlorides, and other halides; the effects of degradation products from separators and wetting agents, and other organic materials used in cell construction; and metallic ions from cell components.

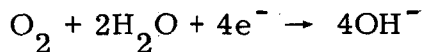
The effects of impurities will be determined by specific ion addition to the cell electrolyte at controlled concentration levels where possible and by the addition of electrolyte which has been previously in contact with a specific component under conditions to promote degradation of the component. For example, the degradation of the separator material would be accomplished by refluxing the separator material in the electrolyte in the presence of oxygen at elevated temperatures for a fixed period of time. The same experiment could be repeated by having a charged positive electrode present during the refluxing to note if it contributes to the degradation process. Similar type experiments could be performed with any of the cell components individually to identify if they are a source of deleterious impurity. At the present time, we intend to restrict the degrading type experiments to the range of 50-60°C and initially to exposure times of 50-100 hours. The resultant electrolyte with the degraded or reaction products would then be added to a test cell for evaluation of its effect on performance. Chemical analysis would be employed to identify components present in the electrolyte and concentrations. The amount of electrolyte added to a test cell would be varied depending on the concentration levels in the sample.

3.0 EXPERIMENTAL EQUIPMENT

During the first Quarter, the experimental apparatus for monitoring oxygen evolution continuously during controlled charge and discharge cycling was installed and tested. Figure 3-1 shows a schematic diagram of the oxygen monitoring system. Basically, nitrogen carrier gas is fed to a manifold system through a Fairchild Stratos precision pressure regulator so that the manifold pressure is held constant in the range of 20-30 psi gauge pressure. The flow rate of the carrier gas through the precision needle valves is essentially independent of minor pressure changes downstream. The gas is saturated with water vapor by bubbling through a solution of potassium hydroxide of the same composition as the test cell electrolyte. The flow rate of the saturated carrier gas is measured with a soap film flowmeter downstream of the bubbler. The carrier gas picks up oxygen being evolved by the nickel hydroxide electrode in the test cell and carries it to the Beckman oxygen sensor.

The current required to maintain a 0.8 v potential across the Beckman oxygen sensor cell is a function of the partial pressure of O_2 in the cell environment. Figure 3-2 is a schematic drawing of the cell and auxiliary equipment to illustrate the operating principles. Carrier gas is directed into the cell housing where some oxygen diffuses through the Teflon membrane to the sensing cell (the amount of oxygen diffusing through the membrane is a function of the permeability of the membrane--a constant--and the partial pressure of oxygen in the carrier gas. The latter is a function of the gas flow rate and the rate of oxygen generation). The oxygen is reduced at the gold cathode while the silver anode is oxidized according to the reaction:

Cathode Reaction



Anode Reaction



The cell potential must be held at 0.8 v for these reactions to occur. When the partial pressure of oxygen is about 160 mm (~21%) the current

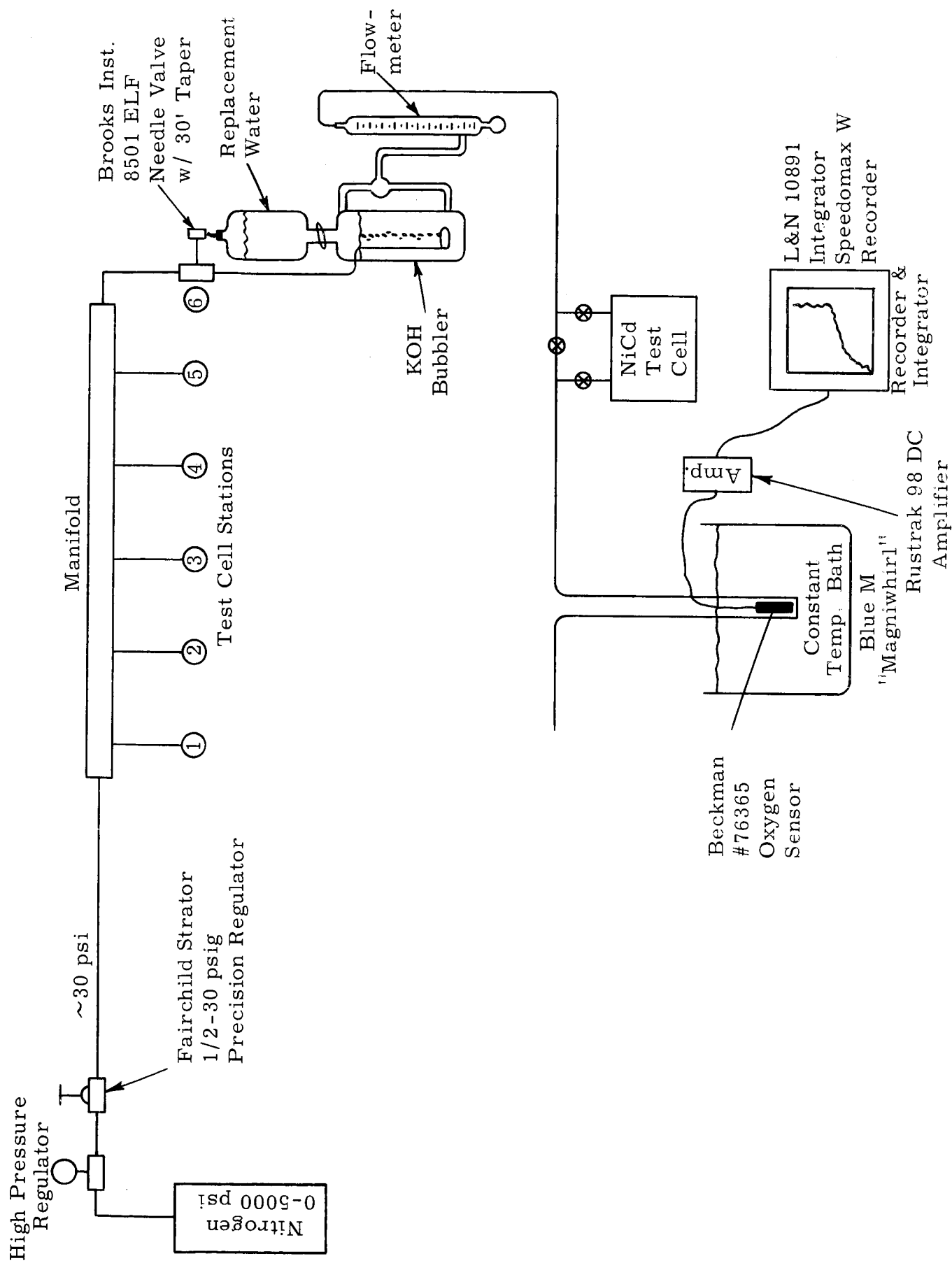


Figure 3-1. Oxygen Monitoring System Schematic.

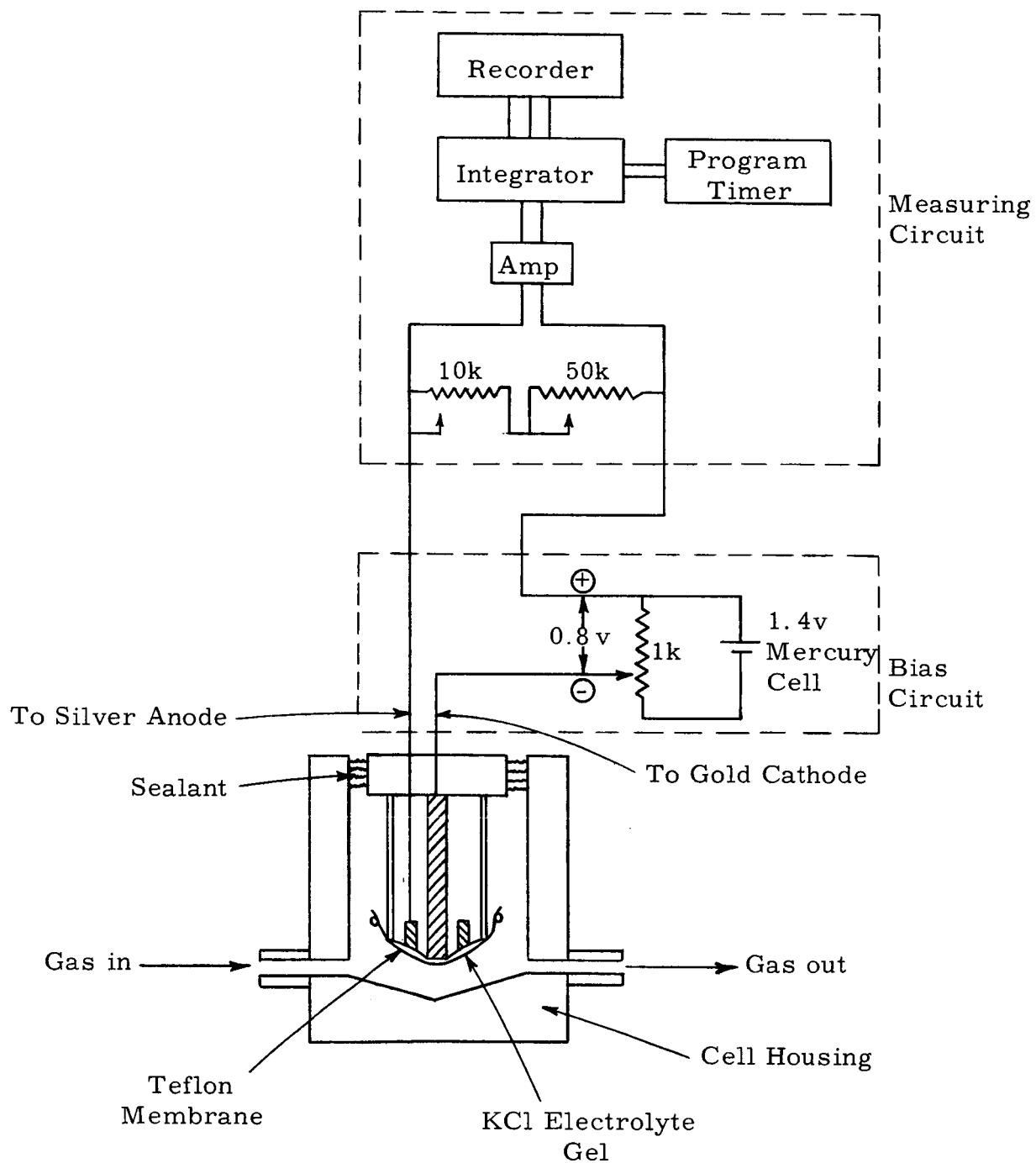


Figure 3-2. Beckman Oxygen Sensing Cell - Schematic.

consumed in the cell is approximately 0.8 microamperes. The amplifier-recorder combination shows a maximum deflection for a ten millivolt signal. The resistors of the measuring circuit must be approximately 1.2 kilohms, where a 20% concentration of O_2 is the maximum anticipated. This value varies from one cell to another, of course, and must be set and calibrated separately for each channel. A constant temperature bath must be used with the monitoring cells because of the temperature coefficient of oxygen permeation in the Teflon membrane.

The nitrogen flow rate should be set, however, so that the maximum concentration of oxygen will not exceed 10% so that the differential of voltage change with respect to oxygen partial pressure change will remain essentially constant over the course of the experimental run. Low flow rates of carrier gas (~ 5 cc/min) are indicated to determine the onset of gassing while higher flow rates (~ 20 -40 cc/min) will be required to monitor total gassing behavior. Assuming the voltage recorded on the recorders is directly proportional to partial pressure of oxygen in the gas stream, the effect of flow rate on the recorded potential at different gassing rates may be seen in Figure 3-3.

An additional complicating factor is that the input impedance of the Rustrak 98DC Amplifier is 1 megohm. Therefore, the resistance of the current measuring portion of the oxygen sensor cell circuit is limited to about 100,000 ohms to preserve scale linearity. This in effect limits the lower sensitivity of the oxygen detection to about 0.05% oxygen in the carrier gas (when 0.2 mv on the recorder is assumed to be the lowest reading discernible from "noise" on the recorder). At low carrier gas flow rates (~ 5 cc/min) this corresponds to the 1 ma gassing rate.

Figure 3-4 is a schematic representation of the test cell used in this system.

An alternate method of monitoring gassing rate uses a Niedrach-Alford type electrode in conjunction with charged cadmium counter electrodes to consume the oxygen as fast as it is generated and monitoring the resulting current. This requires a modified top for the test cell shown in Figure 3-4. Figure 3-5 is a representation of the oxygen sensing modification and the

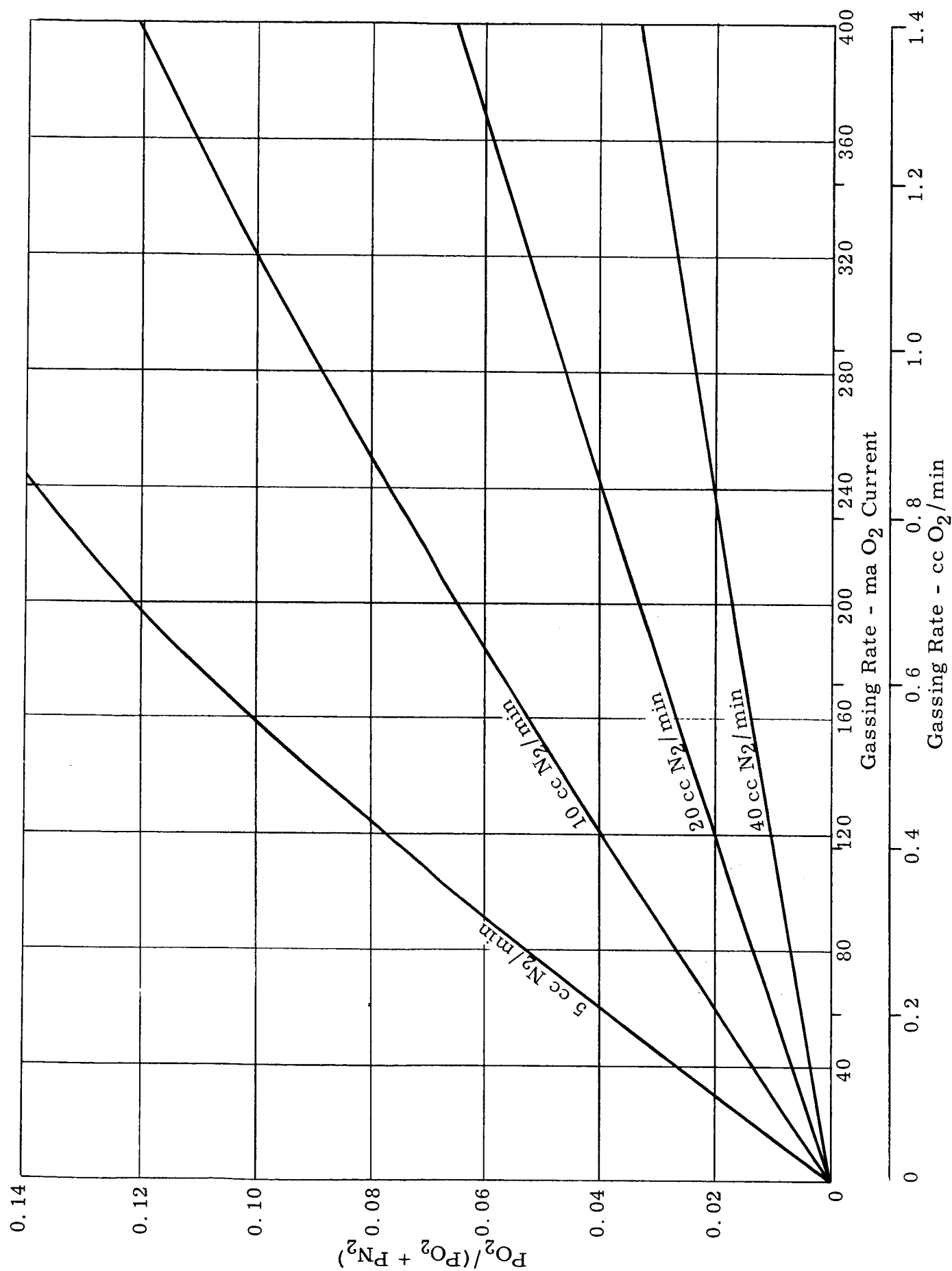


Figure 3-3. Partial Pressure of Oxygen in Carrier Gas at Different Flow Rates.

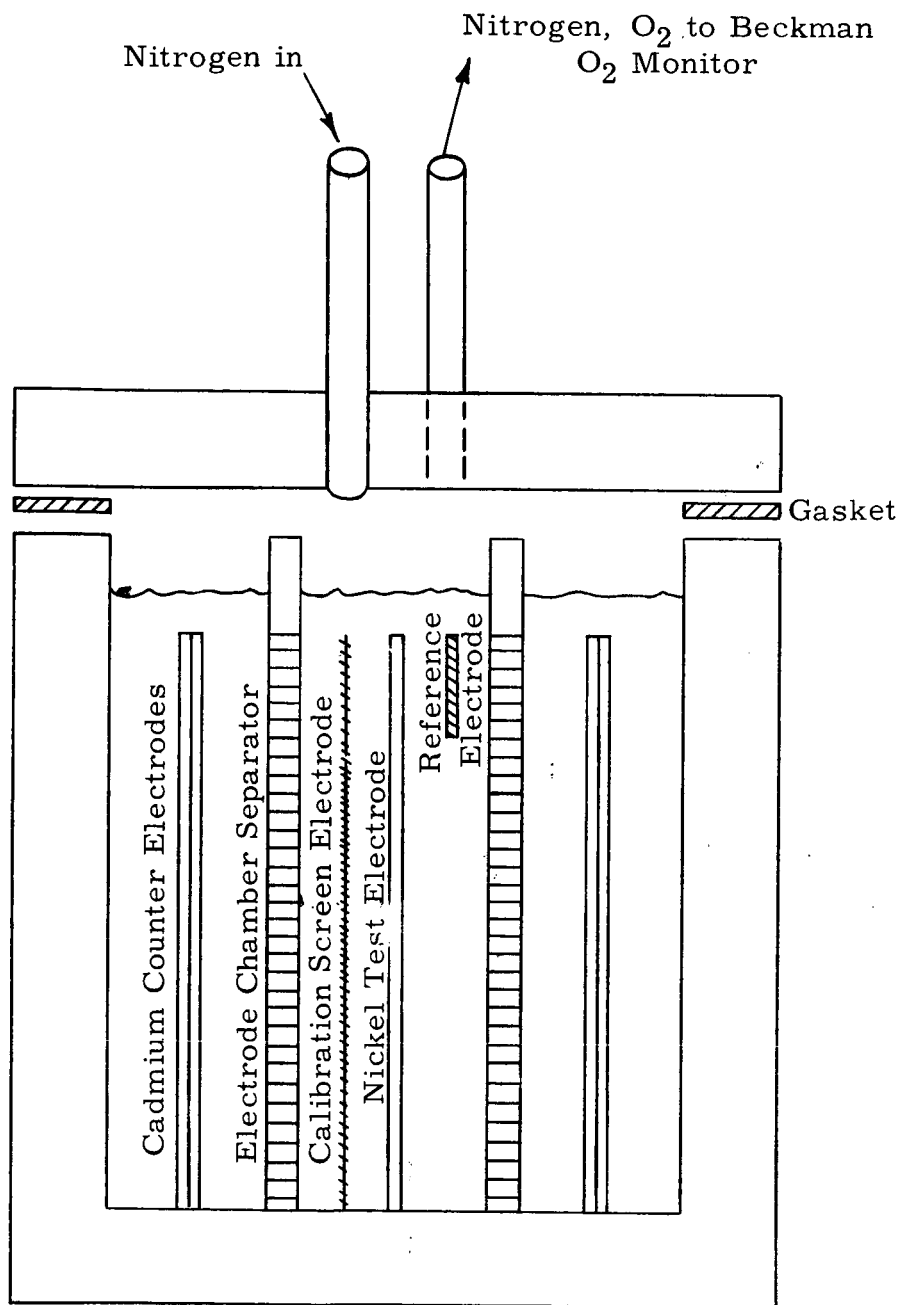


Figure 3-4. Oxygen Monitoring Test Cell.

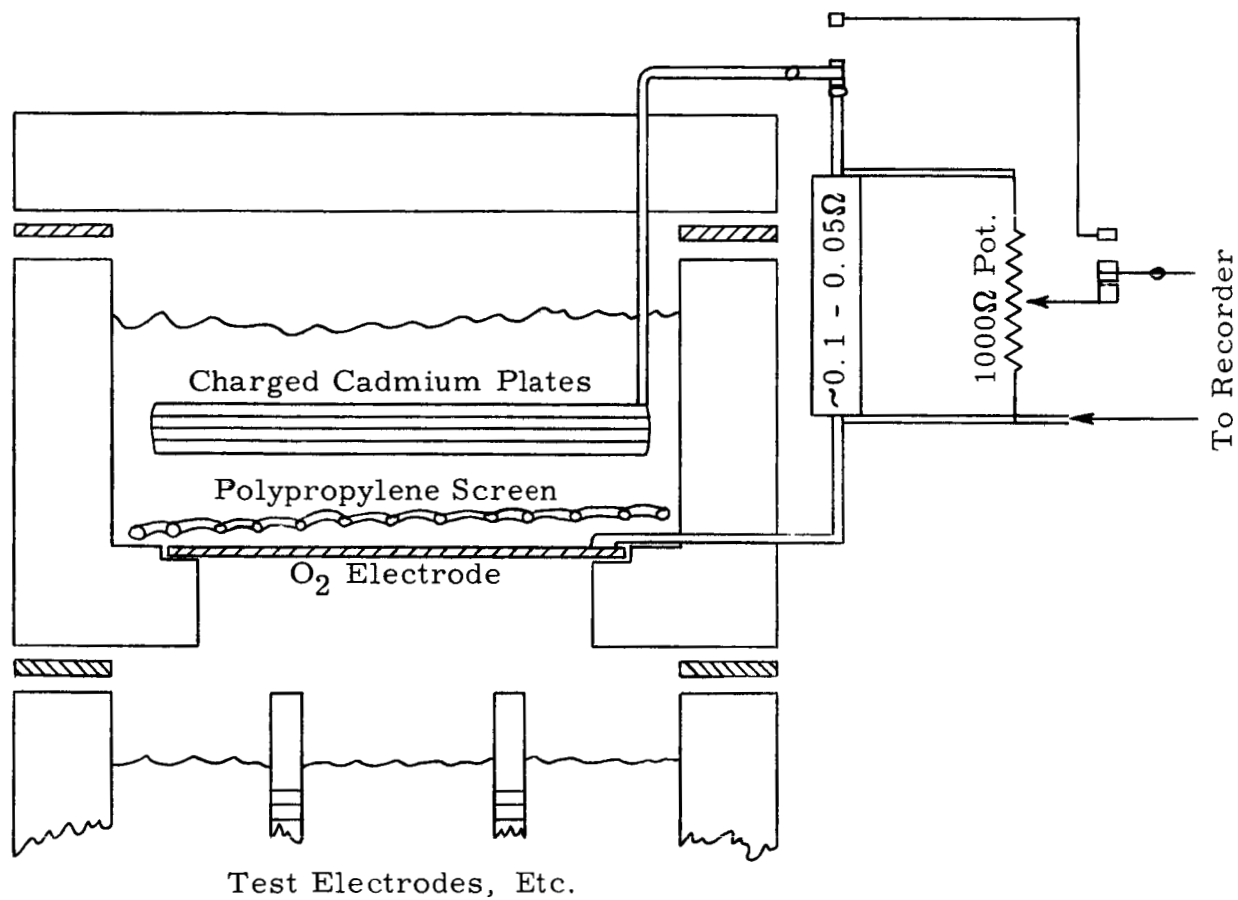


Figure 3-5. Alternate Oxygen Monitoring Test Cell.

required electrical circuit. Use of an auxiliary relay switch can change the sensitivity from approximately 5 ma (when full scale deflection is 250 ma) to direct reading in microamperes gassing current if desired. Such low gassing rates, however, would be difficult to observe because of the solubility of the gas in the electrolyte and the slow diffusion in the test cell. Nevertheless the sensitivity of this technique is sufficiently greater, that it warrants further consideration.

Figure 3-6 shows a part of the six station oxygen monitoring facility. The oxygen test cell in the center with the bubblers and flowmeters for five stations in the background. The bubblers will be raised above their present position when the test program is underway. The cell on the left is a prototype of the alternate test cell.

Figure 3-7 shows the test traces of gassing determination trial runs performed using the two cells shown in Figure 3-6. The top recorder is from the Beckman oxygen sensing electrode while the bottom trace used the alternate design. Chart speed for both recorders was 12 in/hr while the carrier gas flow rate was about 50 cc/min. Oxygen was evolved from a nickel screen in each cell which were connected in series. The "noise" level is about the same in each trace but the response time is noticeably faster in the alternate design. The nonlinearity of the latter was due to heating of the current measuring resistor which was insulated during this test.

The cyclic testing equipment for controlled charge and discharge of test cells, and the characterization equipment for individual plates used in the previous program are available and will be used in this program. Schematic diagrams for this equipment may be found in Appendix I of the Final Report Contract NAS 5-3477, December 31, 1964, Characterization of Nickel-Cadmium Electrodes.

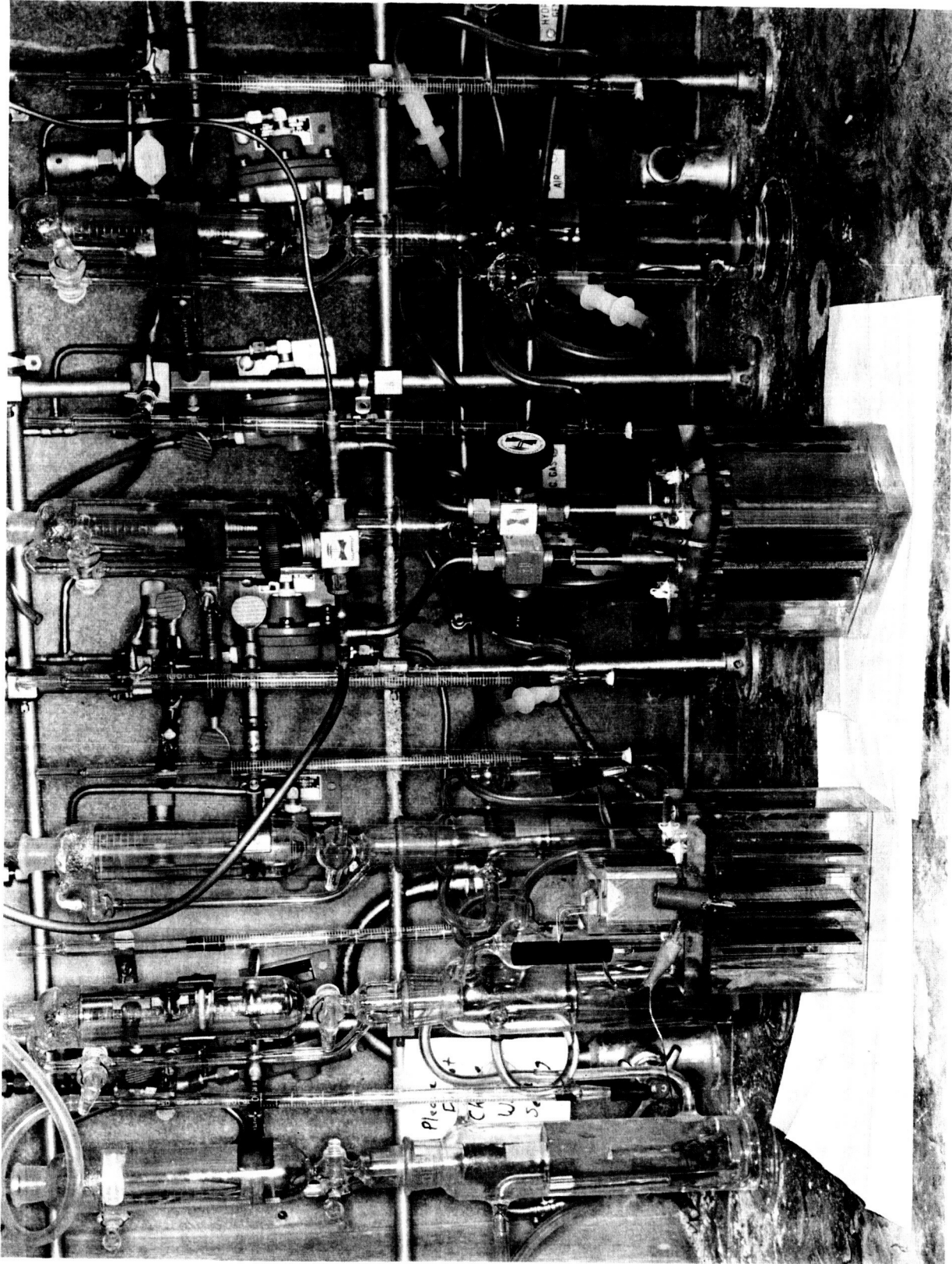


Figure 3-6. Oxygen Monitoring Facility

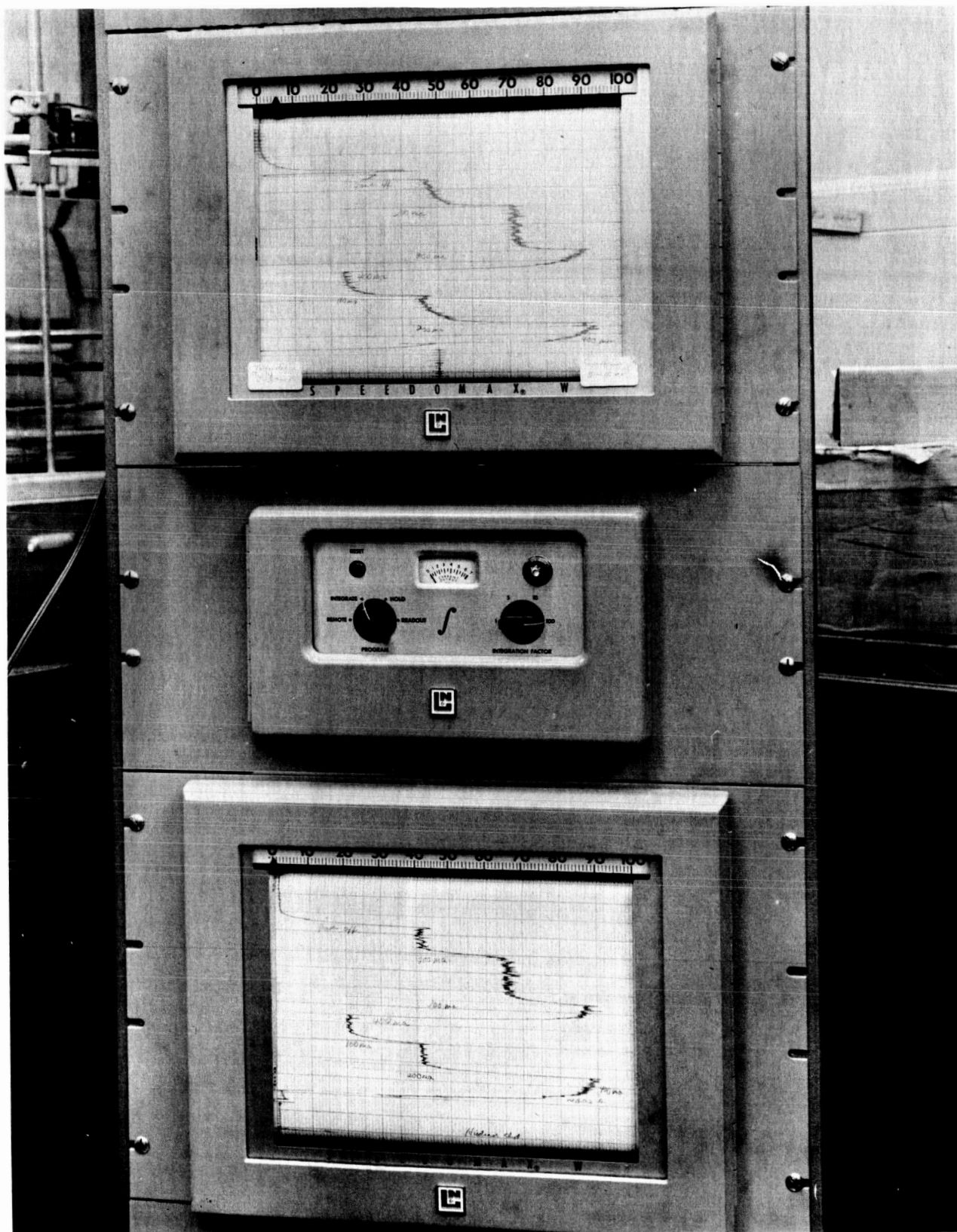


Figure 3-7. Oxygen Rate Test Traces

4.0 PROGRAM FOR NEXT QUARTER

Plates for this program will be received the last week of September. Experimental work will start October 1. The majority of the work during the next quarter will be to characterize the individual plates to determine the capacity distribution for the lot. Towards the end of the quarter after sufficient plates have been characterized, work on the positive electrode behavior studies will be started.